Standard Operating Procedure for GLNPO Board Analyses

LG500

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TABLE OF CONTENTS

Section Number	Subject	Page
1.0	SCOPE AND APPLICATION	1
2.0	SUMMARY OF METHOD	1
3.0	SAMPLE HANDLING AND PRESERVATION	1
4.0	INTERFERENCES	1
5.0	APPARATUS	1
6.0	REAGENTS	2
7.0	INSTRUMENT CALIBRATION	4
8.0	ANALYTICAL PROCEDURE	6
9.0	CALCULATIONS	7
10.0	QUALITY CONTROL	7
11.0	PREVENTIVE MAINTENANCE	11
12.0	TROUBLESHOOTING/CORRECTIVE ACTION	12
13.0	SAFETY AND WASTE HANDLING	12
14.0	REFERENCES	12
15.0	WARNING AND CONTROL LIMITS	13

STANDARD OPERATING PROCEDURE FOR GLNPO BOARD ANALYSES

1.0 SCOPE AND APPLICATION

1.1 This method is a description of the procedure used on the EPA Monitoring vessel, the *R/V Lake Guardian,* and includes the determination of Specific Conductance, Total Alkalinity as $CaCO_3$, Turbidity and pH.

1.2 Dissolved Oxygen, when determined, is analyzed at nearly the same time as the subject analyses, but is described in another SOP.

2.0 SUMMARY OF METHOD

2.1 At the beginning of each lake cruise, applicable instruments, used for the water quality analyses of lake water samples, are initially calibrated and control standards analyzed.

2.2 For routine sample analyses, a sample is placed on the specific conductivity apparatus and heated to 25.0°C. The conductivity reading is recorded, after which the pH electrode is placed in the sample and that reading is recorded. The beaker is removed from the apparatus, and the content is used to fill the turbidity cuvette and the 100-mL alkalinity sample volume measuring flask. The alkalinity flask is transferred to the alkalinity apparatus and the titration is performed. The readings from the alkalinity titration and the turbidimeter are recorded.

2.3 A set of control standards is run after the last station sampled on each 12-hour shift.

3.0 SAMPLE HANDLING AND PRESERVATION

3.1 Samples are collected in new one-gallon cubitainers (polyethylene plastic collapsible containers) (See *Field Sampling Using the Rosette Sampler*).

3.2 Analyses are routinely performed within two hours of collection. The cubitainers are retained refrigerated for the duration of the 12-hour shift, so that, if control standards show a system to be out of control, it can be brought back into control and the samples re-analyzed for that parameter(s) (See WQS QAPP).

4.0 INTERFERENCES

4.1 No major interferences are known with regard to these analyses with Great Lakes water samples.

5.0 APPARATUS

5.1 Conductivity Meter, with nominal l-cm cell constant (YSI Model 35 with YSI probe 3401).

5.2 Variable speed stirring motor with glass stirring paddle. Two each.

5.3 Immersion heater with on/off controller.

5.4 Thermometer with 0.01°C subdivisions, range 0/30.

5.5 Meter pH (Fisher Accumet Model 15) with Ross combination Electrode and automatic temperature compensation two each meter and electrode.

5.6 Turner Designs Model 100 Nephelometer.

5.7 Buret, 25-mL automatic fill to zero, or Digital valveless burette.

5.8 The conductivity apparatus consists of: the Meter with a conductivity electrode; a small electric motor, with a speed controller, driving a stirring paddle; a thermometer with 0.01°C divisions; a small table to hold the sample beaker such that the thermometer, the electrode, the stirring paddle, and the immersion heater are immersed in the sample.

5.9 The alkalinity apparatus consists of: a pH meter and electrode; a small electric motor with a speed controller, driving a stirring paddle; a burette; and a small table to hold the sample beaker such that the electrode and stirring paddle are immersed in the sample with the burette tip draining into the sample beaker.

6.0 REAGENTS

6.1 Reagent water: All reagents are prepared using water that has passed through two ion exchange cartridges, an organic removal cartridge and a 0.2-micron filter. The specific conductance of this reagent water used for standard preparation is less than 0.l µmhos/cm (10 megohm-cm). The feed water to this purification system is from a polyethylene tank fed from a 'Super Still' (a vacuum distillation system maintained by the engineering staff).

6.2 Conductivity

6.2.1 Stock sodium chloride set standard solution. 10.000 mg of NaCl (dried at 105°C for two hours) is dissolved in reagent water and diluted to one liter in a volumetric flask.

6.2.2 Working Calibration Standards

6.2.2.1 The following are prepared with volumetric labware. Two liters of each of these standards are prepared prior to the survey. A calibration standard wash will be maintained for each of the calibration standards the same as is done with the control standards.

mL of 10 gm/L NaCl diluted to 1 liter	Specific Conductance µmhos/cm
20	415.8
15	313.5
10	210.3
5	106.1

6.2.3 Stock Conductivity Control Standard Solution: 10.000 mg of KCl (dried at 105°C for two hours) is dissolved in reagent water and diluted to one liter in a volumetric flask.

6.2.4 Control Standards: The following are prepared using volumetric labware.

QC Type	mL 10.00 g/L KCl diluted to 1 Liter	Specific Conductance µmhos/cm at 25°C
High Check Standard (CH)	15	293.3
Low Check Standard (CL)	10	196.5

6.2.4.1 Three gallons of each of these working control standards are prepared prior to the survey.

6.3 Turbidity

6.3.1 Formazin working standards

Prior to each survey, two solutions are prepared. Shelf life is one month.

1.00 mg/100 mL hydrazine sulfate, $(NH_2)_2H_2SO_4$
10.00 mg/100 mL hexamethylenetetramine

6.3.2 The two solutions are brought to 25°C and five mL of each are mixed in a 100-mL clean dry volumetric flask. After 24 hours at 25°C, the mixture is diluted to 100 mL. Shelf life is one month. Nominal turbidity is 400 NTU.

6.3.3 Four dilutions of stock 400 NTU are prepared for calibrating the turbidimeter according to 7.4. The instrument is calibrated at the beginning of each lake survey, i.e., Michigan spring 2000, Huron spring 2000. Shelf life 24 hours.

Aliquot of 400 NTU diluted to 100 mL with reagent water	Final NTU value
5 mL	20
2 mL	8
0.5 mL	2
0.1 mL	0.4
Reagent Water	0

6.3.4 The control standards for turbidity are obtained from APS Analytical Standards. They are 0.5 NTU and 10.0 NTU.

6.4 pH

6.4.1 Calibration standards for pH. Commercial pH standards (10, 7 and 4) are used for the pH standardization.

6.4.2 Commercial buffer packets (6.86 and 9.18) are used to prepare the pH check standards.

6.5 Preparation of 0.02 N sulfuric acid solution.

6.5.1 Commercial standard 1.00 N sulfuric acid is diluted to 20 mL per liter with reagent water.

6.6 Alkalinity

6.6.1 Na_2CO_3 • 0.1 N. The contents of a Dilutit Commercial Standard capsule are diluted to one liter or a commercial 0.1 N solution is used or prepared from a 1 N standard.

6.6.2 Total Alkalinity Control Standards

Concentration	mL/L of 0.1 N Na_2CO_3
100 mg/L T.A. as $CaCO_3$	20
40 mg/L T.A. as $CaCO_3$	8

7.0 INSTRUMENT CALIBRATION

7.1 Conductivity

7.1.1 The same YSI Model 35 conductivity meter and electrode have been used by this program for more than 5 years without calibration. The high and low control standards have been within an acceptable range throughout that period.

7.1.2 Prior to the first and second lake of each survey, the calibration standards are measured at 25°C and the results recorded. If any of the standards produce results on the second standardization that differ by more than 1 μmho/cm from the first standardization, the series is measured again just prior to the next lake, until each standard from two consecutive series differ by no more than 1 μmho/cm.

7.1.3 The nominal value of the conductivity cell is 1, but may not be exactly 1. If the values from the calibration deviate from the actual values of the standards, a correction factor must be derived to apply to the control standard readings prior to evaluating whether the control standards are within range. The average standard/reading for the four calibration standards should be recorded and applied to the control standards. Enter the correction factor and the derived value in the remarks column. The derived value must fall within the control standard range.

7.2 pH

7.2.1 This paragraph describes the use of the AB15 pH meter for day to day use. To initially set up the meter go to the operation manual for these instruments. The meter for pH determination is standardized at the beginning of each shift with buffers 7 and 10. In the following description, stable readings should be obtained before proceeding to the next step. Each step is numbered. Standardize at the beginning of each shift.

1. Sequence the electrode through lake water, buffer 7 wash, and buffer 7 standard.
2. Record the pH and temperature of the buffer 7 standard. At this point, the meter should be displaying the pH icon, icons for buffers 7 and 10, and the pH should be displayed to two decimal places.

3. Press "setup" twice. "Clear buffer" should be displayed to the left of the beaker icon, "710 " to the right.
4. Press Enter. The area where the buffer icons are displayed should be blank. The uncalibrated value of the pH 7 buffer is displayed.
5. Press STD. The buffer group 2 4 7 10 12 should be displayed briefly.
6. Press STD again. The buffer icon "7" should appear in the buffer icon area. The pH reading should change to the pH 7 buffer value at the temperature of the standardization.
7. Sequence the electrode through lake water, buffer 10 wash, and buffer 10 standard.
8. Press STD. The pH of the pH 10 buffer is displayed assuming an efficiency of 100%.
9. Press STD again. The actual electrode efficiency should be displayed briefly and a 10 should join the 7 to the right of the beaker icon.

Unlike the previous meter, the electrode efficiency is not retained during standardizations. Thus, after a one-point standardization, the meter uses 100 as the efficiency until a second standard is entered, regardless of the actual value.

If the buffers are not cleared prior to standardization, "ELECTRODE ERROR" may appear as result of a slight drift in the electrode potential. This is not an indication that the electrode is bad. It just means that the instrument must be standardized using the proper SOP.

7.2.2 To initially set up the AB15 or AR15, consult the instrument operations manual.

7.3 Alkalinity

7.3.1 The Model 15 ACCUMET pH Meter used for the Alkalinity. Titration is calibrated as above, except that pH buffers 4 and 7 are used instead of 7 and 10.

7.4 Turbidity

7.4.1 The turbidimeter zero is set prior to standardization by the upper screw inside the sample compartment. The zero reading should be set between 0.01 and 0.05 with the cuvette compartment empty, since the meter will not display negative readings. A commercial 20-NTU standard in a separate cuvette (without being agitated) is then used to set the calibrate knob so that the meter reads 20.0. The calibrate knob is not changed after that. Standardization is performed using freshly prepared (from 400-NTU concentrate) 20, 8, 2, 0.4 and 0.0 NTU. The values obtained for the five standards are recorded, and later used to convert the sample readings to corrected values. Samples with turbidity readings over 20 NTU are diluted as necessary to obtain readings and the dilution factor is recorded under remarks. The corrected reading is recorded as turbidity (i.e., reading of the diluted sample x the dilution factor).

8.0 ANALYTICAL PROCEDURE

8.1 General considerations

8.1.1 To preserve the integrity of the sample, the Cubitainer must be agitated before withdrawing an aliquot.

8.1.2 Rinsing of the conductivity equipment or the alkalinity volumetric flask is not necessary between samples from the same lake water station.

8.1.3 The pH measuring electrode should be maintained in lake water except during measurements.

8.1.4 It is not necessary to rinse the 4.5 pH solution off the alkalinity equipment between titrations.

8.2 At each station:

1. A fresh lake water sample is placed on the conductivity apparatus and the temperature is raised to 25°C. This water is then used to rinse the alkalinity volumetric flask and to fill the receptacle for the pH electrode.
2. A fresh aliquot is then poured into the conductivity beaker which is then placed on the conductivity apparatus and temperature raised to 25.0°C while the stirrer is operating to produce rapid circulation without breaking the surface of the sample.
3. When the temperature and conductivity readings are stable at 25.0°C, the conductivity is recorded, and the pH electrode is inserted in the sample.
4. When the pH meter indicates that the pH reading is stable, the pH reading is recorded, and the pH electrode is returned to the pH electrode receptacle.
5. The beaker is removed from the conductivity apparatus and portions are used to fill the alkalinity sample volume measuring flask and the turbidity cuvette.
6. The 100 mL from the alkalinity volumetric flask is placed on the alkalinity apparatus and the stirring speed is adjusted to achieve rapid circulation without breaking the surface of the sample. Titration to pH 4.5 (plus or minus one drop) is performed. The total alkalinity (burette reading x ten (10)) is recorded.
7. The turbidity is recorded as soon as the reading becomes stable.
8. Subsequent lake water samples from the same station are treated as from step 3 above.

8.3 When the apparatus is not being used, the conductivity cell is immersed in a lake water sample. The pH electrode is immersed in a lake water sample. The alkalinity pH electrode is immersed in the last titrated sample. The turbidity cuvette is filled with reagent water. For extended periods of non-use, see 11.1.

9.0 CALCULATIONS

9.1 The specific conductance is read directly from the meter.

9.2 The pH is read directly from the meter.

9.3 The alkalinity is 10x the burette reading.

9.4 The turbidity is recorded directly from the meter. Correction for offset and non-linearity are made when the readings are entered into the database.

10.0 QUALITY CONTROL

10.1 During sampling, High and Low Control Standards are analyzed after the last station on each 12-hour shift for each of the four parameters. If any standard exceeds the warning range, the sample values and control values are recorded, but the system is not used again until the parameter is re-standardized and the control standards have verified that the system is in control. If any standard exceeds the control limit, the parameter is re-standardized and all samples analyzed since the last valid control standards are re-analyzed along with the control standards. Appropriate information is inserted in the remarks column of samples associated with control standards beyond the control limits or beyond the holding time. If after re-standardization, the control standards are not within normal range, corrective action must be taken before proceeding with further analyses. All analyses (of samples and controls) are recorded regardless of whether the control standards are within limits or not.

10.2 Conductivity

10.2.1 Each conductivity standard consists of unused standard and a bottle holding the ‘wash.’ Each time a standard is to be used, the ‘wash’ is placed on the apparatus and the stirrer is activated to allow the apparatus to equilibrate with that wash. That wash is then discarded and a fresh aliquot of standard is placed on the machine. After the calibration or measurement, this material is then placed in the ‘wash’ container for use as a wash the next time the standard is used. Do not put anything in the standard bottle, do not return ‘wash’ to the wash bottle, do not discard standard. Do place used standard in the wash bottle.

10.2.2 The following QC samples must be prepared and analyzed at the minimum frequency indicated.

QC Type	Frequency	Acceptance Criteria (µmhos/cm)
High Check Standard (CH)	At the onset, starting with the initial calibration of instruments for each lake survey, and after the last station on each 12-hour shift	291 - 296 (warning) 290 - 297 (control)
Low Check Standard (CL)	At the onset, starting with the initial calibration of instruments for each lake survey, and after the last station on each 12-hour shift	194.5 - 198.5 (warning) 193.5 - 199.5 (control)
Field Reagent Blank (FRB)	One per basin[a]	Less than 1 µmho
Field Duplicate (FD1)	One per basin[a]	Difference < 2.0

[a] A field duplicate, lab duplicate, and field reagent blank are collected with each group of 3, 4, or 5 stations depending on the lake. A Random Number Generator (RNG) is used to determine the stations and depths of these QC samples. Where basins are well defined, at least one of each is collected from each basin.

10.3 Alkalinity

10.3.1 The sample volume measurement device for the alkalinity determination must be rinsed with 10 to 20 mL of the appropriate standard or blank prior to use for measuring volume of standard or blank. The titration vessel should never be rinsed with standard or sample. After the pH electrode for the alkalinity is standardized, the electrode should be thoroughly rinsed with reagent water. Thereafter, the titration vessel need not be emptied after each titration until the next use. Neither the electrode nor the vessel needs to be rinsed before the next use, since it contains pH 4.5 material. If the endpoint is overshot, then both the electrode and vessel should be rinsed with reagent water prior to the next attempt. The sample volume measurement device need not be rinsed between samples from the same lake water station, but it should be rinsed with about 20 mL of sample before use for the first sample.

10.3.2 The following QC samples must be prepared and analyzed at the minimum frequency indicated.

QC Type	Frequency	Acceptance Criteria (mg/L)
High Check Standard (CH)	At the onset, starting with the initial calibration of instruments for each lake survey, and after the last station on each 12 hour shift	98 - 102 (warning) 97 - 103 (control)
Low Check Standard (CL)	At the onset, starting with the initial calibration of instruments for each lake survey, and after the last station on each 12 hour shift	38.5 - 41.5 (warning) 38 - 42 (control)
Field Reagent Blank (FRB)	One per basin[a]	Less than 3
Field Duplicate (FD1)	One per basin[a]	Difference < (1 + 0.01 x mean reading)

[a] A field duplicate, lab duplicate, and field reagent blank are collected with each group of 3, 4, or 5 stations depending on the lake. A Random Number Generator (RNG) is used to determine the stations and depths of these QC samples. Where basins are well defined, at least one of each is collected from each basin.

10.4 pH

10.4.1 The electrode for pH determination is maintained in a vessel of lake water. The calibration and control standards for pH are retained in 125-mL bottles labeled with the pH value and either standard or wash. At the beginning of each lake survey, i.e., 'Michigan spring 2000', the ‘wash’ is discarded and the ‘standards’ become the new ‘wash’. Fresh material is then used for the ‘standards’. Each time a standard is used, the electrode is immersed in the ‘wash’ prior to immersion in the ‘standard’. After a ‘standard’ is used, the electrode should be rinsed by immersion in lake water or reagent water. If the vessel that is used for electrode retention is used for rinsing between standards, it should be filled with fresh lake water before the next lake sample determination. Do not attempt to measure the pH of reagent water except in the alkalinity determination. While the true total alkalinity end point for reagent water in not 4.5, we will use that as the end point for simplicity and to be consistent.

10.4.2 The following QC samples must be prepared and analyzed at the minimum frequency indicated.

QC Type	Frequency	Acceptance Criteria (SU)
High Check Standard (CH)	At the onset, starting with the initial calibration of instruments for each lake survey, and after the last station on each 12-hour shift	9.18 ± 0.2 (warning) 9.18 ± 0.3 (control)
Low Check Standard (CL)	At the onset, starting with the initial calibration of instruments for each lake survey, and after the last station on each 12-hour shift	6.86 ± 0.2 (warning) 6.86 ± 0.3 (control)
Field Duplicate (FD1)	One per basin[a]	Difference < 0.3

[a] A field duplicate, lab duplicate, and field reagent blank are collected with each group of 3, 4, or 5 stations depending on the lake. A Random Number Generator (RNG) is used to determine the stations and depths of these QC samples. Where basins are well defined, at least one of each is collected from each basin.

10.5 Turbidity

10.5.1 When taking measurements, the outside of the turbidity cuvette must be clean and dry. It is never touched with bare hands. To preclude spilling water on the outside of the cuvette, the alkalinity sample volume measuring flask can be used to fill the cuvette. The turbidity cuvette cap is marked with an arrow on the lid to allow the cuvette to be placed in the instrument in a repeatable position. The arrow is directed toward the back of the instrument when taking readings. All readings, with the exception of the 20-NTU reference cuvette, should be taken using the same cuvette. It is not necessary to rinse the turbidity cuvette between lake water samples, except where there is a pronounced difference between two samples. When measuring the control standards, the cuvette used for the analyses is rinsed with half a volume of a commercial-grade turbidity standard stored in another cuvette. This rinse solution is discarded. The cuvette used for the analyses is refilled with the standard remaining in the storage cuvette. The cuvette used for the analyses is then filled to capacity with the same commercial standard from a standard bottle. After the analyses, the standard in the cuvette is transferred back to the empty storage cuvette for future use.

10.5.2 The following QC samples must be prepared and analyzed at the minimum frequency indicated.

QC Type	Frequency	Acceptance Criteria (NTU)
High Check Standard (CH)	At the onset, starting with the initial calibration of instruments for each lake survey, and after the last station on each 12-hour shift	10 ± 1.4 (warning) 10 ± 2 (control)
Low Check Standard (CL)	At the onset, starting with the initial calibration of instruments for each lake survey, and after the last station on each 12-hour shift	0.5 ± 0.2 (warning) 0.5 ± 0.3 (control)
Field Reagent Blank (FRB)	One per basin[a]	Less than 0.15
Field Duplicate (FD1)	One per basin[a]	Difference < (.1 + 0.1 x mean reading)

[a] A field duplicate, lab duplicate, and field reagent blank are collected with each group of 3, 4, or 5 stations depending on the lake. A Random Number Generator (RNG) is used to determine the stations and depths of these QC samples. Where basins are well defined, at least one of each is collected from each basin.

10.6 Corrective Action

10.6.1 Corrective action procedures will often be handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, calibration mixes, instrument sensitivity, and any other potential sources of error. If failure occurs and an error is identified, the analyst should re-run quality control and RFS samples in the entire analytical batch to confirm the results. Because analysis of field duplicates and lab duplicates usually occurs after leaving a specific station and re-sampling is largely impossible, re-analysis of these samples to confirm results may be the limit to corrective actions when all other QC samples within a batch meet acceptance criteria. For analyses conducted onboard, if the problem persists or cannot be identified, the matter must be referred to the Chief Scientist for further investigation. Depending upon the Chief Scientist's evaluation, the analyst may or may not be required to re-run the samples. Once a decision is made, full documentation of the corrective action procedures and assessment of the final result must be filed with the WQS QM Technical Lead (Marvin Palmer) or the GLNPO QM.

10.7 Data Reporting/Recording

10.7.1 When corrective actions are not feasible or do not resolve QC failure, the analyst is responsible for identifying all failed QC samples and RFS samples. The analyst should document the QC information on the hard-copy Field Information Recording Forms (Appendix H).

11.0 PREVENTIVE MAINTENANCE

11.1 During surveys, the pH and conductivity electrodes should be kept immersed. Between surveys, the conductivity electrode should be thoroughly rinsed in reagent water prior to allowing it to dry. The pH electrodes should be immersed in pH 7 buffer between surveys so that the glass membrane remains immersed. The level of KCl solution in the reference electrode should be checked at the beginning of each Lake survey and filled to within ¼ inch of the filling hole.

11.2 The turbidity cell should be thoroughly rinsed with reagent water at the conclusion of each survey.

12.0 TROUBLESHOOTING/CORRECTIVE ACTION

12.1 If the conductivity cell is so dirty that bubbles always form at the top of the cell when a receptacle of water is placed on the apparatus, ethanol or 1 N NaOH may be used to attempt cleaning. Neither should be left on the cell for more than two or three minutes.

13.0 SAFETY AND WASTE HANDLING

13.1 Refer to GLNPO's *Health, Safety and Environmental Compliance Manual* (May 1997, or as amended) and individual instrument procedural operations manuals for specific details on applicable 1) personal health and safety issues; 2) instrumental, chemical, and waste handling procedures; and 3) accident prevention. This applies to all EPA personnel, EPA contractors or federal, state, or local government agencies, and persons who operate or are passengers onboard US EPA GLNPO vessels during all activities and surveys.

13.2 All containers storing reagents, standards, controls, blanks, and wastes used in the laboratory must be properly identified through appropriate labeling and hazard definition.

13.3 Every chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. Please refer to Appendix L in GLNPO's *Health, Safety and Environmental Compliance Manual* (May 1997, or as amended) for more detailed descriptions of the potential risks associated with any chemicals used in this method. It is good laboratory practice to wear a lab coat, safety goggles and gloves at all times.

13.4 It is the responsibility of the user of this method to comply with relevant chemical disposal and waste regulations as sited in GLNPO's *Health, Safety and Environmental Compliance Manual* (May 1997, or as amended). All applicable safety and waste handling rules are to be followed. Good technique includes minimizing contaminated waste.

13.5 Over-board discharges of chemical wastes are forbidden.

14.0 REFERENCES

14.1 "Methods for Chemical Analysis of Water and Wastes," March, 1979. EPA Publication #600/4-79-02.

14.2 "Operating Instructions," YSI Model 35 Conductivity Meter.

14.3 "Calibration of Conductance Cells at 25°C with Aqueous Solutions of Potassium Chloride," April, 1959. Journal of the American Chemical Society. 1557-1559.

15.0 WARNING AND CONTROL LIMITS

Board Parameters 2000

Parameter	QC Check	Value	Warning Limit	Control Limit
pH	High	9.18	± 0.2	± 0.3
pH	Low	6.86	± 0.2	± 0.3
pH	Duplicate	0	0.2	0.3
Conductivity	High	293.3	± 2	± 3
Conductivity	Low	196.5	± 2	± 3
Conductivity	Duplicate	0	1.5	2
Turbidity	High	10	± 1.4	± 2
Turbidity	Low	0.5	± 0.2	± 0.3
Turbidity	Duplicate	0	0.2 + 0.07 (a+b)	0.2 + 0.1 (a+b)
Total Alkalinity	High	100	± 2	± 3
Total Alkalinity	Low	40	± 1.4	± 2
Total Alkalinity	Duplicate	0	1 + 0.007 (a+b)	1 + 0.01 (a+b)